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Indian Standard

METHOD FOR DETERMINATION OF NICKEL BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

METHOD FOR DETERMINATION OF NICKEL BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

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0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 10 July 1987, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 The atomic absorption spectrophotometric method is dependent on the fact that atoms in ground state absorb light of the same wavelength as they emit, when excited. When radiation from a given excited element is passed through a flame containing ground state of atom, the intensity of the transmitted radiation will decrease in proportion to the amount of the ground state of atoms of element in the flame. A hollow cathode lamp whose cathode is made of the element to be determined provides the radiation. The metal atoms to be measured are placed in the beam of radiation by aspirating the specimen into an oxidant fuel flame. A monochromator isolates the characteristic radiation from the hollow cathode lamp and a photosensitive device measures the attenuated transmitted radiation. The absorbance is taken as a measure of concentration of the element analysed.

0.3 Various products where nickel estimation may be required include alloys, steels, ores, minerals, effluents, petroleum products, soils, catalysts, etc.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the atomic absorption spectrophotometric method for determination of nickel.

*Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemical and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. OUTLINE OF THE METHOD

3.1 The sample is dissolved in a mixture of hydrochloric acid and nitric acid, digest and evaporated to syrup, dissolved again in nitric acid (1 : 1) by boiling, allowed to settle and filtered. The filtrate is made to a definite volume. Suitable dilution is aspirated into the air-acetylene flame, making standards in the same way. Nickel in solution can be determined by atomic absorption spectrophotometric method using 232·0 nm wavelength with a slit of 0·2 nm band pass in air-acetylene flame.

3.2 Preparation of Standard Stock Solution — Dissolve 0·250 0 g of metallic nickel of 99 9 percent purity (or A. R. nickel-salts of equivalent amount of metallic nickel) in (1 : 1) nitric acid and heat to dissolve completely. Add few ml of concentrated hydrochloric acid and boil. Dilute to 250 ml. This will give 1 000 µg/ml of Ni (*A*). From this parent stock solution (*A*), prepare a standard of 100 µg/ml solution (*B*) and then prepare 1, 2, 3, 4, 5, 10, 15, 20 µg/ml nickel solution by appropriate dilution of solution (*B*), adding 10 ml of (1 : 3) nitric acid solution.

4. OPERATING PARAMETERS

4.1 The wavelength at which nickel is to be measured is 232·0 nm, the most sensitive line for nickel with a slit of 0·2 nm (or equivalent). The alternative wavelength is 352·5 nm or 351·5 nm with slit width 0·5 nm or equivalent which may be used in case of higher concentration of nickel. The light source is nickel-hollow cathode lamp and the flame type is air-acetylene oxidizing flame (lean blue).

4.2 Working Range

Wave Length nm	*Range* ppm
232·0	1-10
341·5	10-50
352·4	20-80

*Specification for water for general laboratory use (*second revision*).

5. SAMPLE PREPARATION

5.1 Metals and Alloys — A suitable quantity of the sample is dissolved in (1 : 1) nitric acid or *aqua regia*, evaporated to dryness, again dissolved in hydrochloric acid, diluted, filtered and made-up to a known volume. A suitable dilution is made for determination of nickel, aspirating in the flame.

5.2 Water and Effluents — Nickel content in water is usually at a very low level. Concentration is required before aspirating into flame. Concentration may be done by evaporation in water bath with dilute nitric acid. Evaporated mass is taken up with a mixture of nitric acid and hydrochloric acid diluted, filtered, made-up to a known volume and aspirated into the flame.

5.3 Minerals, Ores, Silicates, Sea-Sediments, etc — Finely powdered samples are digested in a mixture of hydrochloric acid and nitric acid and evaporated to syrupy mass. Perchloric and hydrofluoric acid are added and evaporated to the fumes of perchloric acid. Cool the mass and take in hydrochloric acid solution, dilute, filter if required, and make up to known volume. Original or a suitable dilute solution is aspirated into the flame.

5.4 Food Products, Clinical Samples, Organic Materials, Plant Materials, etc — The sample is brought into solution either by wet digestion with a mixture of nitric acid and perchloric acid in bomb calorimeter or by carefully dry ashing at 450-500°C and dissolving in nitric acid. Filter and make up the filtrate to suitable known volume.

5.5 Atmospheric Particulates — Membrane filter containing particulates is leached in nitric acid and filtered. The solution is made to a known volume.

5.6 Coal and Coke — After ashing at about 550°C, it is dissolved in *aqua regia*, filtered and diluted to a known volume.

6. PROCEDURE

6.1 Transfer appropriate quantity of the material, depending on the amount (percent) of nickel present, in a 400 ml beaker. Add 25-30 ml concentrated nitric acid, warm and then add 30 ml of (1 : 1) hydrochloric acid. Digest to decompose the material and evaporate to syrup. Dissolve in 25-30 ml of (1 : 1) nitric acid, heat to boiling and filter to remove insoluble substances. Make up to a definite volume for 1 000 µg/ml solution of nickel, prepare suitable dilute solution, as required.

6.2 Optimize response of the instrument by adjusting instrumental parameters and flame adjustment. Aspirate water to get zero absorption. Aspirate blank, standard solutions (at least 4) and record the absorption. Prepare calibration curve accordingly, from concentration versus absorbance.

6.3 From the linear portion of the calibration graph, the concentration of nickel in the unknown solution is obtained from the absorbance of the unknown solution. Alternatively, the concentration of nickel in unknown solution may be obtained directly if the apparatus has the built in micro-processor unit or is coupled with DCR unit or computer, after presetting in concentration made with the standard nickel-solution before aspirating the experimental solution.

7. SPECTRAL INTERFERENCE

7.1 With multi-element lamps containing iron, an interference may occur when using the 232·0 nm nickel line if nickel is being determined in an iron matrix. A secondary nickel line (for example, 352·4 nm or 341·5 nm) may be chosen to avoid interference.

7.2 To avoid non-atomic species, if any at the particular wave length, back ground correction, may be used.

NOTE 1 — Single element hollow cathode lamp is preferred over the multi-element hollow cathode lamp.

NOTE 2 — It is always preferable to use more sensitive resonance wavelength than the other alternative lines.

NOTE 3 — For measuring higher concentrations of the element, rotate the burner or dilute the solution appropriately or measure at less sensitive non-interfering line.

NOTE 4 — Use linear portion of the calibration curve for obtaining concentration, if necessary; use curve correction.

8. CALCULATION

$$\text{Nickel, percent by mass} = \frac{C \times V}{10^6} \times \frac{100}{M}$$

where

C = concentration of nickel in µg/ml in final solution,

V = volume in ml of final solution, and

M = mass in g of the sample in final solution.